I hereby certify that this correspondence is being deposited with the U.S. Postal Service with sufficient postage as First Class Mail, in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450, on the date shown below.

Dated: June 11, 2003 Signature:

Keza Mollaaghababa

Docket No.: 101328-0148

Group Art Unit: 1752

(PATENT)

PECEIVED TO 12003 IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of: Theodore H. Fedynyshyn

Application No.: 09/512,942

Filed: February 25, 2000

Examiner: Chu, J.

For: ENCAPSULATED INORGANIC RESISTS

DECLARATION OF THEODORE H. FEDYNYSHYN **PURSUANT TO 37 C.F.R. 1.132**

Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450

Dear Sir:

I, Dr. Theodore H. Fedynyshyn, a citizen of the United States residing at 32 Atkinson Lane, Sudbury, MA 01776, declare as follows:

- 1. I received a Ph.D. degree in Chemistry from Brown University in June 1980.
- I have been employed as a senior scientist at Massachusetts Institute of Technology 2. (MIT) Lincoln Laboratory at 244 Wood Street, Lexington, MA 02420 since October 1997. My responsibilities include conducting and supervising research in the area of resist chemistry, and in particular, in the area of novel resists suitable for use in high energy microlithography.
- 3. I am the inventor of the invention disclosed in the pending United States Patent Application No. 09/512,942 entitled "Encapsulated Inorganic Resists," which was filed on February 25, 2000. I am familiar with the application and the prosecution conducted to date. In particular, I have studied an Office Action issued on June 20,

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2002 in this application and U.S. Patent No. 6,114,083 of Kawamura et al., which was cited in the Office Action.

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4. I have performed experiments on the following formulation of Kawamura described in Col. 31 of Kawamura's patent, and noted in my previous Declaration of November 19, 2002, to determine the base solubility of this formulation upon exposure to actinic radiation as well as in the absence of exposure to actinic radiation.

The experiments described below were conducted to assess the viability of Kawamura's composition as a photoresist. The experiments were designed to simulate actual photolithography and the protocol employed a testing standard commonly used in my laboratory to test actual resist candidates. A suitable candidate for a base soluble positive resist must exhibit essentially 100 percent dissolution in the portions exposed to actinic radiation and very little (or no) dissolution in the other portions while a suitable candidate for a base soluble negative resist must exhibit essentially 100 percent dissolution in the unexposed portions and very little (or no) dissolution in the other portions.

- 5. A resist, herein referred to as Resist A, was prepared based on above formulation via the following steps:
 - a) A copolymer composed of 80 percent sulfonic acid ester and 20 percent hydrolytic polymerizable trialkoxysilane, referred to in Kawamura as copolymer I-11,

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was prepared by free radical polymerization of 80 parts of 1-methyl-2-propyl styrene-4-sulfonate and 20 parts of 3-trimethoxysilyl-propyl methacrylate in tetrahydrofurane.

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- b) A 29% solution of silicon dioxide nanoparticles in ethyl lactate was prepared.
- c) Resist A was prepared by combining 5.4 grams of a 20% solution of copolymer of step (a) in tetrahydrofurane, with 0.36 grams of tetraethoxysilane, and 0.12 grams of a 50% solution of phosphoric acid and stirring the resulting solution for approximately 10 minutes, followd by addition of a solution of 0.06 grams of di(t-butylphenyl)iodonium camphor sulfonate in 4.12 grams of ethyl lactate. The resulting solution was stirred for about 5 minutes and was then filtered through a 1 micrometer fiberglass filter.
- 6. Resist A was spin cast as a 164 nm thick film onto a silicon wafer followed by a post application bake (PAB) at 130 C for about 60 seconds.
- 7. Portions of the applied film was exposed to actinic radiation at a wavelength of 248 nm at a series of different doses ranging from 1 to 96 mJ/cm² while other portions remained unexposed.
- 8. Subsequent to exposure, the wafer was baked at 130 C for about 90 seconds. The wafer was then developed by immersion in a surfactant containing aqueous base developer (2.38% aqueous tetramethyl ammonium hydroxide solution) for about 60 seconds, and was then removed and rinsed with DI water, and dried under a stream of nitrogen gas.
- 9. Resist A did exhibit a latent image after exposure and the baking steps indicating that a chemical reaction had occurred in the exposed portions of the resist.
- 10. Base solubility of both the exposed and unexposed portions of Resist A was determined by comparing the thickness of the applied film subsequent to the above development step in an aqueous base solution with the thickness before the development.

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11. The applied film of Resist A retained approximately 64 percent of its thickness in the unexposed areas, and retained approximately 85 percent of its thickness in the areas exposed to 96 mJ/cm² of radiation, subsequent to the above development step that includes immersion of the wafer in an aqueous base developer for about 60 seconds.

- 12. Resist A did *not* exhibit a minimum base solubility either in the exposed or unexposed portions required for use of Resist A, respectively, as a positive or negative resist at 248 nm.
- 13. Resist A exhibited a decrease in the contact angle of decalan and a decrease in the contact angle of water as the exposure dose was increased from 1 to about 40 mJ/cm², which is consistent with deprotection of the protecting group of polymer. However, this chemical change did not lead to an aqueous base developable film, as noted above.
- 14. Another resist, herein referred to as Resist B, was also prepared based on the above formulation of Kawamura via the following steps:
 - a) 4.8 grams of a 20% solution of the above-described copolymer I-11, composed of 80 percent sulfonic acid ester and 20 percent hydrolytic polymerizable trialkoxysilane, in tetrahydrofurane was combined with 0.36 grams of tetraethoxysilane, and 0.12 grams of a 50% solution of phosphoric acid, and the resulting solution was stirred for about 10 minutes.
 - b) To this solution was added a solution of 0.06 grams of di(t-butylphenyl)iodonium camphor sulfonate and 0.414 grams of a 29% solution of silicon dioxide nanoparticles in ethyl lactate in an additional 4.12 grams of ethyl lactate.
 - c) The resulting solution was stirred for about 5 minutes and was then filtered by a 1 micrometer fiberglass filter.
- 15. Resist B was spin cast as a 88 nm film on a silicon wafer followed by a PAB at 130 C for about 60 seconds.

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16. Some portions of the resist film were exposed to actinic radiation at a wavelength of 248 nm at a series of exposure doses ranging from 1 to about 96 mJ/cm² while other portions remained unexposed.

- 17. Subsequent to exposure, the wafer was PEB baked at 130 C for about 90 seconds.

 The wafer was then developed by immersion in a surfactant containing 2.38% aqueous tetramethyl ammonium hydroxide solution for about 60 seconds. The wafer was then removed and rinsed with DI water, and was dried under a stream of nitrogen gas.
- 18. Resist B did exhibit a latent image after the above exposure and baking steps indicating that a chemical reaction had occurred in the exposed portions of the resist.
- 19. Base solubility of both the exposed and unexposed portions of Resist B was determined by comparing the thickness of the applied film subsequent to the above development step in an aqueous base solution with the thickness before the development.
- 20. The applied film of Resist B retained approximately 80 percent of its thickness in the unexposed areas, and retained approximately 90 percent of its thickness in the areas exposed to 96 mJ/cm² of radiation, subsequent to the above development step that includes immersion of the wafer in an aqueous base developer solution for about 60 seconds.
- 21. Resist B did *not* exhibit a minimum base solubility either in the exposed or unexposed portions required for use of Resist B, respectively, as a positive or negative resist at 248 nm.
- 22. Resist B exhibited a decrease in the contact angle of decalan and a decrease in the contact angle of water as the exposure dose was increased from 1 to about 40 mJ/cm², which is consistent with deprotection of the protecting group of the polymer.

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However, this chemical change did not result in an aqueous base developable film, as noted above.

I further declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

May 27, 2003

Bv

Theodore H. Fedynyskyn

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